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**EUROPEAN PATENT APPLICATION**

(21) Application number: 79200607.4

(51) Int. Cl.<sup>3</sup>: **A 23 L 1/22**  
**A 23 F 5/46**

(22) Date of filing: 22.10.79

(30) Priority: 03.11.78 US 957272

(43) Date of publication of application:  
28.05.80 Bulletin 80 11

(84) Designated Contracting States:  
BE DE FR GB IT NL

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(54) Fluidized bed process for making beverage, food or the like.

(57) Absorption drying provides a means for agglomerating dried food and beverage solids around an aroma/flavor core, thereby essentially encapsulating said core and preserving volatile flavor and aroma compounds. The absorption drying process is especially useful for manufacturing instant coffee. Thus, a viscous liquid extract comprising coffee aromas and flavors is sprayed into a fluidized bed of instant coffee powder. Encapsulation of the coffee extract occurs as multiple particles of the coffee powder attach themselves to the aroma/flavor droplets to form aggregates. Rapidly drying the aggregates preserves the aroma/flavor in the core of the aggregates until released during use.

**EP 0 011 324 A1**



FLUIDIZED BED PROCESS FOR MAKING  
BEVERAGE, FOOD OR THE LIKE

This invention encompasses a process for capturing and retaining aromas and/or flavors of the type found in foods and beverages, and the compositions secured therefrom. The process herein is especially useful in the manufacture of beverages, particularly instant coffee.

Commercial instant coffees are made by aqueous extraction of roast and ground coffee, followed by one of two basic drying processes. The older and less expensive process involves spray-drying the water extract. Unfortunately, volatile aroma and flavor components can be lost during spray-drying, and spray-dried instant coffees are generally characterized as less flavorful and aromatic than roast and ground coffees. Freeze-drying of coffee extracts is a more expensive process, but does generally yield a higher quality product with better aroma retention than spray-dried coffees.

The present invention involves spraying small drops of aroma/flavor concentrates into a fluidized bed of dry coffee solids. Each atomized droplet is coated with dry coffee solids to "lock in" the desirable aromas and flavors. This process is termed "absorption drying" herein.

The absorption drying process of this invention provides a low-cost alternative to freeze-drying. By operating in a concentrated system composed of an aroma-rich core surrounded by a dry shell, the retention of volatiles during absorption drying is the same or better than with freeze-drying. It is to be understood that the adsorption drying process of this invention is not limited to the manufacture of instant coffee but can be used in the manufacture of a wide variety of instant foods, beverages and the like. In particular, instant grain beverages having excellent coffee-like aromas and flavors can be made by the present process, as can instant teas, instant fruit-flavored beverages, instant chocolate beverages, instant soups, and the like.

Hair, Cody and McLain, U.S. Patent 3,615,669, issued 1971, disclose a fluidized bed agglomeration process for manufacturing coffee.

5 U.S. Patent 3,903,295, issued 1975, discloses a process for encapsulating materials which tend to degrade within a film-forming agent.

10 Thijssen, H.A.C., "The Effect of Process Variables on Aroma Retention in Drying Coffee Extract", ASIC, June 2-6, 1969; and Thijssen, H.A.C., "Effect of Process Conditions in Drying Coffee Extract and Other Liquid Foods on Aroma Retention", ASIC, June 1973, relate to studies of the mass transport properties of model aroma compounds during a coffee drying operation.

15 Chandrasekaren, S.K. and King, C.J. address the topic of "Volatiles Retention During Drying of Food Liquids", AIChE J., 18, pp. 520-526, 1972.

20 Rulkens, W.H. has considered the "Retention of Volatile Trace Components in Drying Aqueous Carbohydrate Solutions" in his Ph.D. Thesis, Eindhoven University of Technology, 1973.

25 Chandrasekaren, S.K. and King, C.J. discuss diffusion of volatiles in their article "Multicomponent Diffusion and Vapor-Liquid Equilibria of Dilute Organic Components in Aqueous Sugar Solutions", AIChE J., 18, pp. 513-520, 1972.

30 Additional work in the area of the retention of volatiles is reported by Rulkens, W.H. and Thijssen, H.A.C., "The Retention of Organic Volatiles in Spray-drying Aqueous Carbohydrate Solutions", J. of Food Tech., 7, pp. 95-105, 1972.

Kerkhof, P.J.A.M. and Schoeber, W.J.A.H., Advances in Preconcentration and Dehydration of Foods, A. Spicer (ed.), Applied Science Publishers Ltd., London, 1973,

p. 349 and Rulkens, W.H. and Thijssen, H.A.C., Trans. Inst. Chem. Engrs., 47, p. 292, 1969 are relevant to the general topic of volatiles retention. 0011324

Uemaki, O. and Mathur, K.B., "Granulation of Ammonium Sulfate Fertilizer In a Spouted Bed", Ind. Eng. Chem: Process Des. Dev., 15, pp. 504-508, 1976, disclose fluidized bed processes for fertilizer granulation.

The abstraction and capture of volatile aroma and flavor materials from coffee and other edibles such as oranges are disclosed in U.S. Patents 3,595,669; 3,717,472 and 3,997,685, to R.G.K. Strobel. The Strobel procedures can be used to prepare the aroma and flavor concentrates used in the present process.

U.S. Patent 3,704,132, to Strobel, discloses a method for purifying coffee oil for use as a coffee aroma carrier.

Coffee extracts of the type used herein can be prepared by various art-disclosed means, including that described in U.S. Patent 3,700,466, to Bergeran and Schlichter, and in U.S. Patent 3,700,463, to Bolt and Strobel.

U.S. Patent 3,625,704, to Andre, Joffe and Strang, discloses a means for preparing instant coffee in the form of flakes.

U.S. Patent 3,769,032, to Lubsen, Strobel, Reinhart and Patel, discloses a means for preparing aroma-enriched coffee products.

Several U.S. patents disclose means for preparing spray-dried coffee particulates and other instant foods and beverages. See U.S. Patents 2,771,343; 2,750,998; and 2,469,553. See also Sivetz and Foote, "Coffee Processing Technology", Avi Publishing Co., 1963.

The foregoing U.S. patents and references disclose means for preparing various components used in the practice of the present invention, and are incorporated herein by reference.

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Disclosure of the Invention

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The present invention provides a process for manufacturing a water-dispersible composition for use as an instant food, instant beverage, or the like, characterized by:

5 (a) atomizing a liquid aroma concentrate, a flavor concentrate, or a mixed aroma/flavor concentrate, commensurate with said food, beverage or the like, and having a viscosity of from 5 centipoise to about 200,000 centipoise, as droplets into a fluidized bed, the solids in said fluidized bed comprising a multiplicity of particles, or mixtures of particles  
10 and flakes, comprising a dried extract of said food, beverage, or the like, at a ratio of droplets:solids of at least 1:5 by weight, whereupon said particles and flakes form aggregates on said droplets, said aggregates having a moisture content less than about 15%; and

15 (b) drying said aggregates substantially immediately after their formation to a moisture level of less than about 10%, preferably to a level in the range from about 2.5% to about 4.5%, especially from about 3.0% to about 3.5%.

Drying the aggregates quickly, i.e., substantially immediately after their formation and recover, is critical for  
20 securing products having optimal aromas and flavors. Moreover, overall product aroma and flavor are optimized by performing the process under conditions such that a dried shell forms quickly around the aroma/flavor-rich droplet. Aroma and  
25 flavor components are impermeable to the dried shell and thus are retained in the product aggregate throughout the process. These observations are in accord with the reports by Thijssen (above), and reflect the fact that the diffusivity of aroma and flavor in a coffee extract system is dependent on the water  
30 concentration. Thus, at lower water concentrations (ca. < 10% preferably < 3.5%), such as are present in the shell of the dried aggregate, the diffusivity of aroma-type compounds is three orders of magnitude less than the diffusivity of water. Indeed, the relative diffusivity of the aroma volatiles is  
35 so small that the shell can be considered as being selectively permeable only to water. Accordingly, by drying the aggregate products of the present processes as soon as possible after their formation, water is removed before the desirable volatiles can escape.

The present invention also encompasses water-dispersible compositions for use as instant foods, instant beverages, or the like, said compositions being in the form of aggregates, said aggregates comprising:

- (a) a core comprising an aroma concentrate, a flavor concentrate, or, preferably, a mixed aroma/flavor concentrate commensurate with said foods, beverages, or the like, said core being substantially surrounded and encapsulated by;
- (b) a multiplicity of particles, flakes, or, preferably, mixtures of particles and flakes comprising a dried extract of said foods, beverages, or the like, merged on the outer surface of said core.

The process of this invention is especially useful in the manufacture of instant coffee compositions in the form of aggregates, said aggregates comprising:

- (a) a core of coffee aroma or flavor materials, or, preferably, mixtures thereof, said core being substantially surrounded by and encapsulated by;
- (b) a multiplicity of particles, flakes, or, preferably, mixtures of particles and flakes, of dried coffee extract, merged on the outer surface of said core.

Highly preferred coffee, etc., compositions herein are those wherein said aggregates contain no more than about 4.5% by weight of moisture and are free-flowing.

Highly preferred instant coffee compositions herein comprise the coffee aroma/flavor core surrounded and encapsulated by a mixture of dried coffee flakes and coffee particles merged to form an aroma/flavor encapsulating shell. The weight ratio of particles:flakes is in the range of from about 30:70 to about 70:30, generally about 60:40. Such coffee compositions most preferably contain no more than about 4.5% by weight of water, and are free-flowing.

All percentages herein are by weight, unless otherwise specified.

The following describes a preferred and convenient mode of practicing the present invention to manufacture an instant coffee product characterized by improved flavor and aroma qualities. It will be appreciated that the same method can be used to manufacture other instant beverages such as instant tea, instant fruit drinks, and the like.

At the heart of the process is a fluidized bed of spray-dried coffee particles. Alternatively, this fluidized bed can be a mixture of different types of dried solids, for example grain and coffee, to prepare a grain-coffee beverage. The fluidized bed can have the solids in different forms; the solids are preferably a spray-dried instant coffee powder admixed with instant coffee flakes. The presence of the flakes in the final aggregate product greatly improves product appearance and desirably enhances the structural integrity of the aggregates.

Fluidization is a process wherein fine solids are made to behave in a fluid-like manner. Fluidization is accomplished by allowing a stream of gas (e.g., air, nitrogen, etc.) to rise through a bed of coffee particles. Fluidization will take place when the force of the fluidizing gas exceeds the force of the weight and friction factors within the bed of solid particles. A dense phase fluidized bed behaves much like a boiling liquid, so a stream of atomized droplets of concentrated aroma/flavor can be introduced into the fluidized bed of coffee, whereupon the droplets are quickly coated.

A convenient fluidized bed apparatus used to prepare pilot plant batches of adsorption dried instant coffee in the manner of this invention comprises a 15 cm. I.D. vertical column ca. 1.435 m. in height with a Plexiglass viewing section at the lower bed portion and a stainless steel disengaging section. The gas ( $N_2$  or air) distributor for fluidizing the solids is a porous sintered

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metal disc comprising the base of the column. The 0011324  
supply is a 1.27 cm. compressed air or N<sub>2</sub> line supplying  
80-100 psi.

5 A spray nozzle is positioned centrally in the  
sintered disc and is directed upwardly to atomize the  
aroma/flavor liquid directly into the fluidized bed com-  
prising the coffee particles and flakes. The exit port of  
the nozzle protrudes ca. 10.2 cm. into the cylinder; the  
entrance port is connected to a reservoir of the liquid  
10 aroma/flavor concentrate. For an apparatus of the fore-  
going size the nozzle is 508 microns ( $\mu$ )/1270  $\mu$  (I.D./O.D.),  
with the air cap having a 1778  $\mu$  I.D. (Spraying System  
pneumatic nozzle). A nozzle of these dimensions provides  
a full cone 18° spray.

15 In operation, the cylinder is charged to ca. 15%  
of its height with coffee particles or flakes (ca. 0.65  
kg.). Air or N<sub>2</sub> is introduced into the column via the  
sintered metal base to fluidize the bed of coffee. The  
concentrated aroma/flavor solution is then atomized  
20 through the spray nozzle into the dense phase fluidized  
bed of coffee, whereupon the desired instant coffee aggre-  
gates are formed. The aggregates (ca. 10-15% by weight  
water) are removed from the cylinder and dried, preferably  
within 1-2 minutes after removal, and within about 5 min-  
25 utes after their formation, to a moisture level of ca.  
2.5%-4.5%, most preferably ca. 3.0%-3.5%, thereby locking  
in the aroma/flavor in the form of a core for the indi-  
vidual aggregates.

30 It will be appreciated that the concentrated aroma/  
flavor solution used in the foregoing manner should be  
somewhat viscous and tacky so that the solid coffee parti-  
cles and flakes adhere to the atomized droplets. The  
solute content should be as high as possible, consistent  
with achieving the desired viscosity. An aqueous aroma/  
35 flavor solution with a viscosity below about 5 centipoise  
(cps) is not useful in the practice of this invention



since the water with dissolved aroma/flavor solutes simply wicks into the solids and does not form the desired structure comprising the core encapsulated by the shell formed as the particles and flakes merge. The viscosity range is dependent on the dissolved solutes in the aroma/flavor solution, and these solutes generally comprise ca. from about 30% to about 70% of the solution, which corresponds to viscosities in the range of from about 19 cps to about 200,000 cps. Preferably, the solute content is ca. 50%. Preferably the viscosity is in the range of 500-700 cps.

It is highly preferred that the process of this invention be carried out using a mixture of instant coffee particles and instant coffee flakes, for several reasons. First, a better quality of fluidization in the fluidized bed is achieved, presumably due to the broader range of sizes of the solids being fluidized. The presence of shiny coffee flakes in the final agglomerated product also contributes substantially to product appearance. For example, coffee products made in accordance with this invention containing coffee flakes have a rich, brown appearance. Other products made by this process, e.g., orange juice beverage aggregates containing orange flakes, have a desirable sheen. Moreover, the flakes contribute importantly to the stability of the aggregates. During the fluidizing process, the aggregates being formed are continually subjected to abrasion until such time as they are removed from the fluidized bed and dried. It is important that the aggregates not disintegrate, since the aroma/flavor volatiles in their cores would be lost. The presence of flakes in the aggregates helps maintain their integrity during formation. Moreover, the presence of the flakes in the aggregates helps maintain aggregate integrity during the subsequent drying step, which preferably comprises drying the aggregates while they are being vibrated.

The coffee particles used in the practice of this invention comprise spray-dried instant coffees. As manu-

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factured, spray-dried coffee has an average particle size in the 5-300 micron range and the particles are hollow. For use herein, the spray-dried coffee particles are preferably milled (and thereby densified) to a particle size

5 in the 5-50 micron range.

The instant coffee flakes used herein are somewhat larger than the coffee particles. The optimal flakes pass through a 16 mesh Standard Sieve. Flake manufacture is described in U.S. 3,625,704 (above).

10 The aggregation step of this process is carried out by atomizing the tacky liquid aroma/flavor concentrate into the dense phase of the fluidized bed of solid instant coffee. Droplet size of the concentrate is in the 50-500 micron range, that is, slightly larger than the solid bed

15 material. In general, 5 parts by weight of bed solids to 1 part of spray provide excellent agglomerates. The rate of introduction of the concentrate into the dense phase of the fluidized bed affects the rate of agglomeration. In the extreme, if the concentrate is atomized too rapidly,

20 the individual droplets of concentrate coalesce; this is to be avoided. A convenient rate of atomization for preparing instant foods and beverages in a pilot scale apparatus as disclosed above introduces about 24 grams of the liquid aroma/flavor concentrate into the bed per

25 minute. Thus, in a typical pilot-scale bed comprising 650 g. of 1:1 coffee particles and flakes, 130 g. of aroma/-flavor concentrate is introduced over a period of about 5 minutes and 23 seconds.

30 It is highly preferred that the aggregates as prepared in the bed contain no more than about 10% to about 12% total moisture. If the moisture content is allowed to become greater than about 15% (e.g., by using slightly damp coffee solids as the bed material, by using excess water in the aroma/flavor concentrate, or by operating at

35 too high a humidity), the aggregates are too tacky and

begin to set up in a solid mass before they can be recovered from the fluidizing apparatus. As disclosed below, after aggregation is complete, the moisture content is further reduced by a drying step.

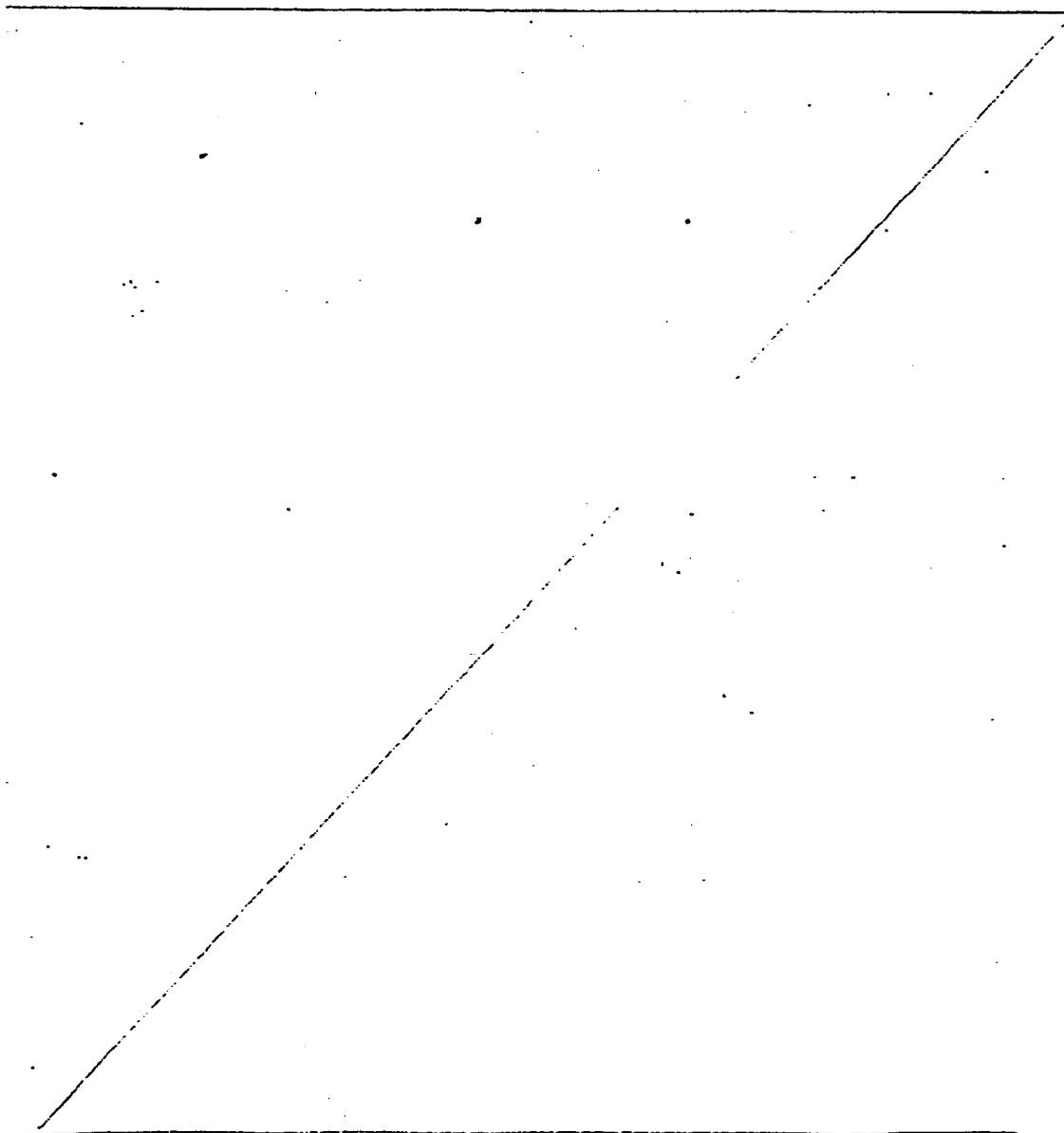
The aggregation step of the present process is conveniently carried out at temperatures of about 20°C-30°C. If the fluidized bed is allowed to become too warm, desirable aroma and flavor volatiles can be lost. If the fluidized bed is run at extremely cold temperatures to minimize the loss of volatiles, longer drying times in the subsequent step are required.

After the aggregates are formed, they are dried. Proper control of the drying step is very important. If after they are formed the aggregates are allowed to stand for a substantial period of time (ca. 3-5 minutes, or, perhaps, somewhat longer, depending on the surrounding conditions of temperature and humidity) the desirable aromatic and flavor volatiles in the core of the aggregates will migrate to the surface and be lost. Accordingly, it is important that the aggregates be dried substantially immediately after they are formed and recovered from the fluidized bed. Again, this can depend somewhat on the moisture content of the core, but for aggregates which comprise about 10%-12% moisture, drying should begin within about 1-2 minutes after the aggregates are recovered from the fluidizing apparatus. Preferably, a continuous fluidized bed apparatus is equipped with a standard collecting device so that, as the aggregates are formed, they are continuously removed and delivered directly to the dryer.

Drying can be carried out in any manner which reduces the moisture content of the aggregates to the desired range quickly. Preferably, the drying time for an individual aggregate is about one minute. Forced air, warm forced air (preferred: 90-100°C air), radiant heat, vacuum drying, and the like, can be used, as long as

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dryness is quickly achieved so that the shell of merged particles around the core "lock in" the volatile aroma/- flavor components. Whatever means of drying is used, it is important to aroma/flavor retention not to allow the temperature of the aggregates, themselves, to exceed about 70-75°C. Preferably, the aggregates are vibrated continuously during the hot air drying step. In an optimal (ca. one minute; 90-100°C) forced air vibratory drying step, the aggregates reach temperatures of about 60°C.. Commercial apparatus such as the Jeffry Vibratory Dryer are convenient for use in the drying step of the present process.



Industrial Applicability

The practice of the present invention is further illustrated by the following examples, which are not intended to be limiting of the type of ingestible compositions which can be manufactured by the present process.

Example I

In accordance with the procedure disclosed in U.S. Patent 3,997,685, a coffee column having a width of ca. 12.7 cm. and a length of 15 cm. is placed in communication with two in-line condensing traps. The first trap is held at  $-76^{\circ}\text{C}$  by dry ice. The second trap is held at  $-195.8^{\circ}\text{C}$  by liquid nitrogen. A vacuum pump is connected to the system to allow the use of vacuum pressures during operation.

900 grams of roasted coffee beans are frozen in liquid nitrogen and ground to a fine grind size, i.e., less than 20 mesh U.S. Standard Sieve. In order to purge the system of oxygen, 10 grams of solid carbon dioxide are placed on the beans and allowed to sublime and be carried through the system to displace oxygen. The roast and ground coffee is placed in the column. Water at  $90^{\circ}\text{--}100^{\circ}\text{C}$  is introduced to produce wet steam which is pulsed downward through the column zone.

The cold wet steam is passed in a downward manner through the column in a pulsing fashion and introduced at approximately two-minute intervals in approximately equal amounts. A black band of materials is evident in the uppermost portions of the column. During the run this band continually moves downward through the column prior to breakthrough. Prior to breakthrough, a colorless frost is collected in the first trap and retained.

The second trap, which is held at liquid nitrogen temperatures, provides a solidified aroma frost which

comprises coffee aroma and carbon dioxide solidified into an aroma-CO<sub>2</sub> matrix.

The melted frost from the two traps prepared in the foregoing manner is admixed with spray-dried coffee solids to prepare a ca. 50% aqueous concentrate of total dissolved coffee solids, viscosity ca. 604 centipoise. The amount of aroma/flavor fluid used can be adjusted according to the needs of the formulation. An aroma/flavor concentrate having a viscosity in the 600-700 cps range is preferred.

The aroma/flavor concentrate prepared in the foregoing manner is atomized into a fluidized bed comprising ca. 60% (wt.) of spray-dried instant coffee powder and ca. 40% (wt.) instant coffee flakes, using the apparatus and technique described hereinabove. Approximately 100 g. of aroma/flavor concentrate per 500 g. of the mixture of powder and flakes is used.

As the aroma/flavor concentrate is atomized into the fluidized bed, the powder and flakes aggregate on the droplets to form aggregates. Said aggregates comprise ca. 10% water. The aggregates are immediately removed from the fluidized bed column and heat-dried (Jeffrey continuous forced air dryer; ca. 95-100°C air; average moisture content of ca. 3.5% by weight).

The instant coffee product prepared in the foregoing manner has a density of ca. 0.3 g./ml. One teaspoonful (1.5 g.) of the product added to 150 mls. of hot water rapidly dissolves to yield a flavorful, aromatic coffee beverage whose organoleptic properties are fully equivalent to freeze-dried instant coffee.

#### Example II

Coffee beans (25% Robusta; 75% Arabica) are decaffeinated with methylene chloride in standard fashion, roasted, ground, extracted and the extract spray-dried and

crushed to ca. 25 microns. A portion of the decaffeinated instant coffee is converted to flakes by roll milling in the manner disclosed in U.S. 3,625,704.

A 1:1 (wt.) mixture of the decaffeinated coffee particles and flakes is placed in a fluidizing apparatus, as disclosed above. An aroma/flavor concentrate (604 cps) is prepared using decaffeinated coffee solids in the manner of Example I. The concentrate is atomized into the dense region of the fluidized bed and aggregates form. The aggregates are removed from the apparatus and substantially immediately dried (hot air) to a moisture content of 3.5%. The product is a flavorful, aromatic decaffeinated coffee.

### Example III

15        Using the aroma/flavor extraction procedure of Example I, peeled oranges are frozen in liquid nitrogen and finely ground to a mesh size of 7. The ground oranges are placed in the extraction column; 25 grams of carbon dioxide are placed in the system and allowed to sublime to  
20        purge the system of oxygen. During extraction, the system is continually flushed in a downward manner with carbon dioxide.

25        400 ml. of liquid flavor concentrate is collected in the first trap subsequent to breakthrough. The band which continually moves downward through the column as pulsing of the wet steam occurs is a bright, very intense orange color. The liquid flavor concentrate has a pleasant orange aroma. The liquid nitrogen trap contains a very pleasant but not especially intense orange aroma-CO<sub>2</sub>  
30        matrix. The aroma and flavor materials thus prepared are combined for further use.

      An orange juice concentrate is spray-dried to a particle size of ca. 100-300 microns and crushed to ca. 50

microns. The orange powder thus obtained is placed in the fluidizing apparatus.

5 The orange flavor/aroma materials prepared above are admixed with concentrated orange juice (ca. 1,000 cps) to provide an orange flavor/aroma concentrate. The flavor/aroma concentrate is atomized into the fluidized bed of dried orange juice powder in the proportions disclosed in Example I. Aggregates form.

10 The orange aggregates are dried within about 3 minutes after their formation to a moisture content of ca. 3%. When added to water (ca. 8.0 g. per 100 g. of water) the aggregates dissolve to provide a pleasant reconstituted orange juice beverage.

15 The process of Example III is repeated with tomatoes, limes, grapefruit, lemons, 1:1 lemon/lime mixtures and chocolate (cocoa), respectively, and excellent instant beverage products are secured.

20 The instant tomato product of Example III is used at a level of ca. 15 g. per 100 g. of hot water to provide a tomato soup.

#### Example IV

Coffee-flavored grain beverages with excellent coffee aroma and flavor are prepared as follows.

25 A. Preparation of Grain Extract Particles. A dry, refined grain extract is obtained from 500 g. of roasted and ground malted barley. The barley is placed into a ca. 12.7 cm. glass column. The glass column is closed with a disc-shaped lid. The lid is equipped with four openings -- a center opening and three additional openings  
30 symmetrically arranged around the center opening. The center opening is used for placing a nozzle above the barley substrate bed. The distance between nozzle opening and substrate bed is 15 cm. Two of the remaining three openings are used for placing thermocouples into and above



the substrate bed for temperature measurements during the run (two openings). The remaining opening is used for connecting a nitrogen line to the column.

5 The column is evacuated to an absolute pressure of 1 mm. A condensing trap is placed between the vacuum pump and the column to condense volatile materials from the barley bed. The condenser is cooled with liquid nitrogen.

10 The volatiles are driven off by pulling spurts of boiling  $H_2O$  through the nozzle. The spurts of hot water are partially transformed into steam (approximately 20% steam transformation at 1 mm. vacuum). The steam transformation also causes a temperature reduction of the steam/water mixture to  $20^{\circ}C$ .

15 The water is soaked up by the substrate, thereby causing the desorption of additional volatiles which are carried by the steam through the substrate bed (steam distillation). The volatiles and steam-distillable volatiles condense in the trap.

20 Addition of subsequent water/steam spurts ( $\sim 10$  ccm. each) onto the substrate pushes the aqueous front deeper into the substrate bed, causing the desorption of additional volatiles, etc. After ca. 13 minutes, a total of 900 ml. of water is introduced onto the substrate bed, at which point the aqueous front reaches the lower end of the  
25 substrate bed. Now, the condenser containing the volatiles and steam-distillable materials is replaced by a fresh condenser. The volatiles are discarded.

30 Addition of subsequent water (spurts) onto the substrate bed causes the aqueous front, which contains water-soluble refined grain extract (pigments, carbohydrates, proteins, inorganic components, etc.) to exit into the new condenser. The new condenser is placed into a dry ice/solvent mixture.

35 After 30 minutes, 1.9 liters of hot  $H_2O$  will have been introduced onto the substrate bed. 1029 ccm. of  $H_2O$ -soluble grain solids are collected in the trap (draw-off

ratio 1:2.05).

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The aqueous malted barley extract is spray-dried, milled, and sieved. The dry refined grain extract exhibits a bland flavor.

In an alternate mode, grain such as roasted rye, wheat or malted barley is extracted with water in a coffee extract column, concentrated and spray-dried to form soluble grain extract particles.

The dried, soluble grain extract particles prepared in the foregoing manner are used to prepare a coffee-flavored instant beverage composition by absorption drying in the manner of this invention by both of the following procedures.

Procedure 1. An aqueous aroma/flavor coffee concentrate prepared in the manner of Example I is atomized into a fluidized bed of the roasted grain particulates prepared as described hereinabove using the apparatus disclosed herein. The ratio of aroma/flavor concentrate to dry grain particles is adjusted so that about 200 mg. of the concentrate per gram of soluble grain particles is used in the absorption drying process. Processing in the fluidized bed apparatus yields aggregates which comprise a tacky, coffee-flavored core surrounded by the soluble grain particulates. Within about 2 minutes of their formation, the coffee-flavored grain beverage aggregates are removed from the fluidized bed apparatus and dried (forced hot air dryer, as disclosed above; average residence time per aggregate ca. 1 minute).

The coffee-flavored grain beverage product prepared in the foregoing manner is added to hot water at a level of about 1.1% and provides a coffee-flavored beverage.

Procedure 1 is repeated by replacing the coffee-flavored extract with viscous aroma/flavor extracts (650 cps) of tea, beef, oranges, cola nuts, and lemons, respectively. The resulting aggregates are suitable for use as instant beverages, soups and foods.



Procedure 2. The coffee-flavored grain beverage product prepared by Procedure 1 provides a quite acceptable beverage, but does not provide all the aroma and flavor characteristics demanded by the most discriminating coffee drinkers. The following procedure provides a highly flavorful and aromatic coffee concentrate which is used to manufacture instant coffee products which are virtually indistinguishable from high quality, 100% coffee beverages.

Preparation of Coffee for Desorption of Aroma and Flavors. Twenty-five kg. of coffee beans are roasted to a photovolt reading of 70 and quenched, first with one liter of water and then with liquid nitrogen. The water quenching is carried out in the roaster ("Probat" roaster). Immediately after water-quenching, the beans are placed in metal drums and shock-quenched with 20 kg. of liquid nitrogen to a temperature of  $\sim -10^{\circ}\text{C}$ . One kg. of powdered dry ice is mixed into the quenched beans before grinding.

The beans are then ground to a coarse grind (instant coffee grind) and then immediately milled on a "Ross" mill with a distance of 88.9 microns (3.5 mils) between rolls. Hydraulic pressure on the rolls is ca. 350 lbs. Speed of the rolls is ca. 100 rpm. Rolled coffee flakes are secured.

Desorbate Column and Ancillary Equipment. A Desorbate Column for capturing aroma and flavor components is assembled and consists of four components:

1. A cylindrical section 60 cm. in diameter and 50 cm. high. Both ends of this section are ground.
2. A top section, dish-shaped with four openings, receiving a nozzle system for steam/water application,  $\text{CO}_2$ -purging, and thermocouples for temperature measurements. A hot water reservoir is connected to the nozzle system. A solenoid valve is placed between the hot water reservoir and the nozzle system.

The top section is placed on a stainless steel ring plate that provides, via two O-rings, a vacuum-tight seal between the cylindrical and top sections. The stainless steel ring also provides a means for lifting the top section for filling and discharging the column.

3. A false bottom, consisting of a 55% perforated stainless steel plate placed between the cylindrical section and the bottom section. Two O-rings provide a vacuum-tight seal between the two sections. Seven layers of cheesecloth are placed on the perforated plate to prevent the flaked coffee from falling through when loading the column.

4. A dish-shaped bottom, equipped with a valve. The valve extends via vacuum tubing to a condensing system. The condensing system is connected to a mechanical vacuum pump. The pump is protected against water vapors with two cryogenic traps. The three column sections, made of "Duran" glass, are held together by metal flanges. The metal flanges are connected to a tubular frame system supporting the entire column setup.

Loading and Purging the Column. The lid section is lifted with a mechanical winch and 20 kg. of rolled coffee flakes prepared as disclosed above are placed onto the false bottom (cheesecloth).

The rolled coffee flakes are spread out to obtain a level bed surface in parallel with the false bottom plate. The lid is then lowered onto the cylindrical section and the air above the coffee bed is displaced with CO<sub>2</sub>.

Preparation of Aroma/Flavor-Frost Condensing Vessels

The condensing system consists of three 8-liter cylindrical flasks (18.7 cm. diameter) placed in parallel between the Desorbate Column and the vacuum source. The condensing vessels are placed into liquid nitrogen. Each condensing

vessel is coated on the inside with 300 grams of a 60% solution of "Desorbate" coffee base extract obtained from a previous run (180 grams Desorbate base dissolved in 120 ml. of distilled water). The concentrate is poured into the cylindrical vessels and the vessels are then rotated in a near horizontal position until an even coating of concentrate is achieved on the inside walls. The vessels are then quickly placed into the liquid nitrogen to freeze the even coating.

Evacuation of Column. After displacing the air from the column and connecting the condensers to the column on the one side and to the vacuum pump on the other side, the vacuum pump is turned on. The bottom valve of the column is slightly opened to prevent the highly volatile materials from rushing through the condensing system. Evacuation of the column under these delayed conditions takes about 10 minutes.

Desorption and Condensing of Aroma/Flavor Frost. After evacuating the column to 1 Torr, the solenoid valve is opened by an electrical switch for approximately one second. One-half liter of water from the reservoir is pulled into the nozzle system. The nozzle system produces an even spray pattern over the coffee bed. The vacuum in the column causes part (~ 20%) of the hot water (99°C) to transform into steam. The transformation into steam is accompanied by a temperature drop of the steam/water mixture from 99°C to 20°C.

The water is immediately soaked up by the rolled coffee flakes and in turned desorbs volatile gases like  $\text{CO}_2$  and coffee aroma/flavor compounds from the rolled coffee flakes. The evaporation of the volatiles can be visually observed by the formation of gas bubbles at the interface between the wetted part and the dry part of the bed of rolled coffee flakes.

As soon as the formation of gas bubbles subsides, another spurt of hot water is introduced onto the column

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bed. This spurt drives the interface deeper into the coffee bed. The interface develops without any channeling or uneven migration into the coffee bed.

As soon as the rolled coffee flakes are saturated with water, dissolution of soluble materials into the aqueous phase occurs, as evidenced by the formation of a dark brown band of solubles. This dark brown band moves deeper into the coffee bed and renewed activity of volatiles desorption occurs when additional spurts of water are applied. The subsequent Table gives detailed information on various parameters like temperature in and above the coffee bed, the vacuum at the interface and above the column bed, the quantities of water applied, the time elapsed, etc.

Table I  
Various Desorbate Parameters

	Time (Min.)	Head Vacuum (Torr)	Temperature °C			Hot Water Added (liters)	Remarks
			Above	Top of Bed	Bottom		
20	000	1	2	-7	-7	00.0	) CO <sub>2</sub> -purging
	007	40	30	30	15	08	
	010	40	36	36	36	10	
							) Collection of
							) Aroma/Flavor
							) Frost
25	015	80	46	46	46	14.5	) Break Through
	025	100	50	50	47	22.5	
	030	100	46	50	46	23.0	
	040	90	46	45	42	5.0	) Collection of
	055	90	52	50	49	33.0	
30	075	100	50	48	47	39.0	) Collection of
	085	90	49	48	47	42.0	
	090	95	49	49	47	46.0	
	110	90					)

The volatiles consist of CO<sub>2</sub>, aroma/flavor compounds desorbed from the rolled coffee flakes, and steam. The volatiles produce a whitish-slightly yellow layer of aroma/flavor frost in the condensers. (The condensers are

protected against light during collection of the aroma/ flavor frost.)

As shown in Table I, the collection of the aroma/ flavor frost is finished after 30 minutes run-time and application of 23 liters of hot water through the nozzle system. At this time, the interphase begins to "break through" the layers of cheesecloth. At this point the aroma/flavor frost traps are removed from the column system.

The traps are sealed with cellulose plugs and kept in the dark in liquid nitrogen jackets until use. Each of the three aroma/flavor frost traps contains about 400 g. of frost.

Preparation of Coffee Aroma/Flavor Concentrate.

Approximately 340 g. of soluble coffee solids are added to each trap to yield a coffee aroma/flavor concentrate containing ca. 50% total coffee solute (including coffee solids used to coat the traps).

The aroma/flavor concentrate prepared in the foregoing manner is atomized into a fluidized bed of soluble grain particles. The aggregates which form are quickly dried to a moisture content of ca. 3.5%, and are free-flowing. A 1% aqueous solution of the aggregates provides an excellent "coffee" beverage.

The aroma/flavor concentrate of Example II is substantially caffeine-free and can be used in the foregoing manner to manufacture a decaffeinated instant grain-based coffee.

In an alternate mode, a grain-type instant coffee composition can be prepared using a mixed bed of soluble grain and coffee solids. Moreover, the core of grain beverage compositions can comprise mixtures of coffee and roasted grain aroma/flavor components.

As can be seen from the foregoing, the process of this invention provides a convenient means for manufacturing water-dispersible compositions for use as instant

foods, instant beverages, or the like, comprising atom-  
izing an aroma concentrate, a flavor concentrate, or a  
mixed aroma/flavor concentrate commensurate with said  
food, beverage, or the like, as droplets into a fluidized  
5 bed, said fluidized bed comprising a multiplicity of  
particles, flakes, or mixtures of particles and flakes,  
comprising a dried extract of said food, beverage, or the  
like, whereupon said particles and flakes form aggregates  
on said droplets; followed by drying said aggregates sub-  
10 stantially immediately after their formation. In this  
fashion are prepared various instant soup (e.g., beef,  
lamb, pork, mutton, and poultry, especially chicken and  
turkey), instant beverage, etc., compositions which  
rapidly dissolve in water.

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CLAIMS

1. A process for manufacturing a water-dispersible composition for use as an instant food, instant beverage, or the like, characterized by:

5 (a) atomizing a liquid aroma concentrate, a flavor concentrate, or a mixed aroma/flavor concentrate commensurate with said food, beverage or the like, and having a viscosity of from 5 centipoise to 200,000 centipoise, as droplets into a fluidized bed, the solids in said fluidized bed comprising a multiplicity of particles, or mixtures of  
10 particles and flakes, comprising a dried extract of said food, beverage, or the like, at a ratio of droplets:solids of at least 1:5 by weight, whereupon said particles and flakes form aggregates on said droplets, said aggregates having a moisture content less than 15%; and

15 (b) drying said aggregates substantially immediately after their formation to a moisture level of less than 10%.

2. A process according to Claim 1 characterized by allowing said particles, flakes, or mixtures of particles  
20 and flakes to substantially surround and encapsulate said liquid droplets, prior to drying.

3. A process according to Claim 1 or 2 characterized in that said mixture of particles and flakes is at a weight ratio of flakes:particles in the range from 30:70 to  
25 70:30:.

4. A process according to any of Claims 1 to 3 characterized in that the flakes have diameters in the range of from 0.5mm to 12.7mm.

5. A process according to any preceding Claim characterized  
30 in that the particles or flakes comprise a dried, water-soluble grain extract.

6. A process according to Claim 5 characterized in that the grain extract is a malted barley extract.

7. A process according to any preceding Claim characterized  
35 in that the drying step comprises hot air drying.

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8. A process according to Claim 7 characterized in that the hot air drying step further comprises forced air vibratory drying.
9. A process according to Claim 8 characterized in that the forced air vibratory drying is conducted for about one minute at a hot air temperature of from 90°C to 100°C.
10. A process according to any preceding Claim characterized in that the aggregates are dried to a moisture level of from 2.5% to 4.5%.



European Patent  
Office

# EUROPEAN SEARCH REPORT

0011324  
Application number:  
EP 79 20 0607

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl.)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
DX	<u>US - A - 3 615 669 (E. HAIR)</u> * Claims 1-9; column 4, lines 26-68; examples 2,3 * --	1,10	A 23 L 1/22 A 23 F 5/46
	<u>FR - A - 1 561 964 (PROCTER &amp; GAMELE)</u> * Abstract 1; page 6, column 1, paragraph 3 - column 2, paragraph 1; example 1 * --	1,10	
	<u>US - A - 3 989 852 (E. PALMER)</u> * Claims 1-7; examples 4-5 * --	1,2	TECHNICAL FIELDS SEARCHED (Int. Cl.) A 23 L 1/22 A 23 L 1/221 A 23 F 5/46 A 23 F 5/48 A 23 F 5/38 A 23 F 3/40 A 23 F 3/42 A 23 P 1/00 A 23 L 2/28
	<u>DE - B - 1 029 802 (KREBS)</u> * Claim 1; example 1 * --	1	CATEGORY OF CITED DOCUMENTS X: particularly relevant A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention E: conflicting application D: document cited in the application L: citation for other reasons S: member of the same patent family. corresponding document
	<u>FR - A - 2 164 618 (D.E.J. INTERNATIONAL RESEARCH)</u> * Claims 1,2; examples I and II * --	1,5-10	
	<u>US - A - 3 391 003 (K. ARMSTRONG)</u> * Claims 1,19; example 4 * ----	1	
The present search report has been drawn up for all claims			
Place of search The Hague	Date of completion of the search 08-02-1980	Examiner DESMEDT	

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